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Copolymers based sensor to detect and pinpoint milk leaks in pipelines of lacteous process factories

Sensor a base de copolímeros para detectar y localizar fugas de leche en tuberías de fábricas de proceso lácteo

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Resumen

Palabras clave:
Fugas; leche; sensores; polímeros.

Los copolímeros poli(acrilato de n-butilo) al 70% y poli(ácido acrílico) al 30% se prepararon mediante polimerización radicálica en emulsión. El polímero resultante se utilizó en un proceso de coextrusión para fabricar un prototipo de sensor tipo cable formado por alambre de nicromo como núcleo y alambre de cobre envuelto en espiral (bobinados 1.2 cm/ciclo). Se diseñó un circuito de sistema sensor para la detección de fugas de leche. La estructura polimérica mejoró las propiedades de procesamiento de los sensores de alambre con forma distribuida y disminuyó la sensibilidad a la humedad ambiental, evitando falsas alarmas. En pruebas simuladas, el sistema detecta la localización de la fuga con alta precisión (niveles de error de +/- 1%).

Abstract

Keywords: Leaks; milk; sensors; polymers.

Copolymer poly(n-butyl acrylate) and poly(acrylic acid), 70% and 30%, respectively, were prepared by radical emulsion polymerization. The copolymer obtained was used in a coextrusion process to make a sensor cable prototype formed of nichrome wire as core and copper wire wrapped spirally (windings 1.2 cm/cycle). A sensing system circuit was designed for milk leak detection. The polymer structure improves the processing properties for distributed shaped wire sensors and decreases sensitivity to the environmental humidity, avoiding false alarms. In simulated tests, the system detects the leak localization with high accuracy (error levels as +/- 1%).

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Introduction

Milk is a staple food with high consumption in the market; therefore, there is a large industry that supplies it. Global milk production in 2023 reached 965.7 million ton (Food and Agriculture Organization of the United Nations [FAO], 2024). In México, in 2021 the production was 450 million liters per year (Gallegos-Daniel *et al.,* 2023). This industry is highly integrated, its processes range from milk harvesting to packaging. Hence, milk goes through a number of intermediate steps where it is transported by pipelines (Figure 1), resulting in a high probability of leakage generation (Lohr & Rose, 2003). Such leaks not only cause the loss of the product but they also represent a source of contamination, exposing milk to microorganisms (Goff, 2007). This is why it is important to have a distributed sensor that can detect and pinpoint milk leaks at different points of the pipelines, from the reception to the packaging.

Leak detection is being widely researched, generating new technologies (Neeraj *et al.*, 2017). In addition to detection, the rapid and accurate location of the leak has been the subject of intense research (Casillas *et al.*, 2015; Cheng *et al.*, 2017; Penza *et al.*, 2001). There are several methods to locate the site or the leak, for example: leak signature space (LSS) (Casillas *et al.*, 2015), negative pressure wave (NPW) (Jia *et al.*, 2014), and acoustic-based leak detection (Cardell-Oliver *et al.*, 2015). Some sensors may be located within the process, providing real-time data and information (Mendoza-Payán *et al.*, 2009); on the other hand, some sensors can be integrated in the same cable that constitutes them. Cheng *et al.* (2017) developed a ferrofluid filled coaxial cable FPI sensor for magnetic field measurement. Penza *et al.* (2001) developed a surface acoustic sensor wave sensor using Pt-polymer-base as active film. The formation of the polymer that constitutes the cable coating is important because it must possess the qualities to fulfill the purpose for which it was designed. For example, humidity modifies the electrical conductivity and some physical properties of the polymer; therefore, this can be used to detect the location of the leak (Cheng *et al.*, 2017; Mieloszyk *et al.*, 2018).

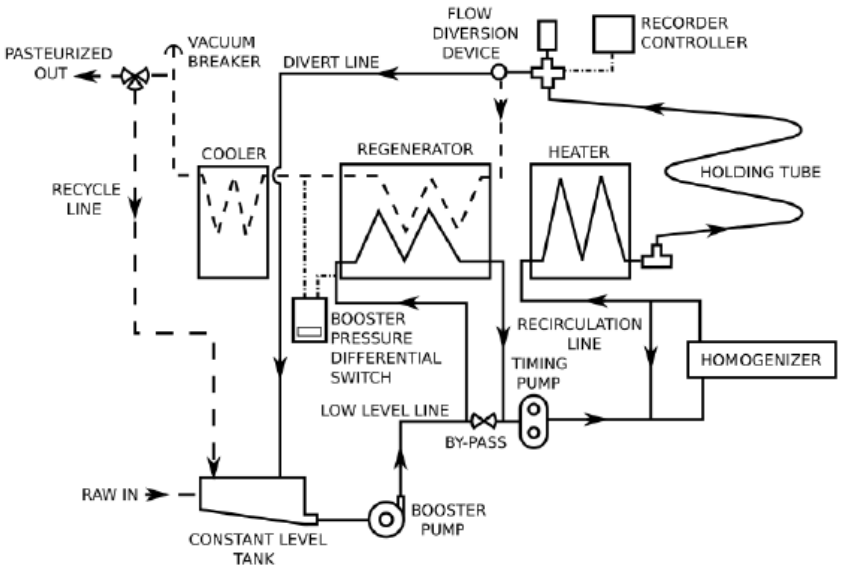

Figure 1. Milk pasteurized process steps.
Source: Author's own elaboration based on Goff (2007).

In this work, a distributed sensor to detect milk leaks is presented. This sensor has a cable form and is sensitive enough to detect relatively small leaks (centiliter per minute) that would go unnoticed by the traditional methods of leak detection (*e.g.*, the mass balance). It is important to note that the authors of the present work have developed several distributed sensors for aqueous solutions, with ultra-fast response (Mendoza-Payán *et al.*, 2009) for water leaks, using crosslinked polyvinyl amine with Cu(II). Following this line of research, the current sensor with a new copolymer of acrylic acid-butyl acrylate has been developed, which improves the processing properties for distributed shaped wire sensors and decreases sensitivity to the environmental humidity, avoiding false alarms (Mendoza-Payán *et al.*, 2009). Figure 2 shows the main features of the sensor disclosed in this paper.

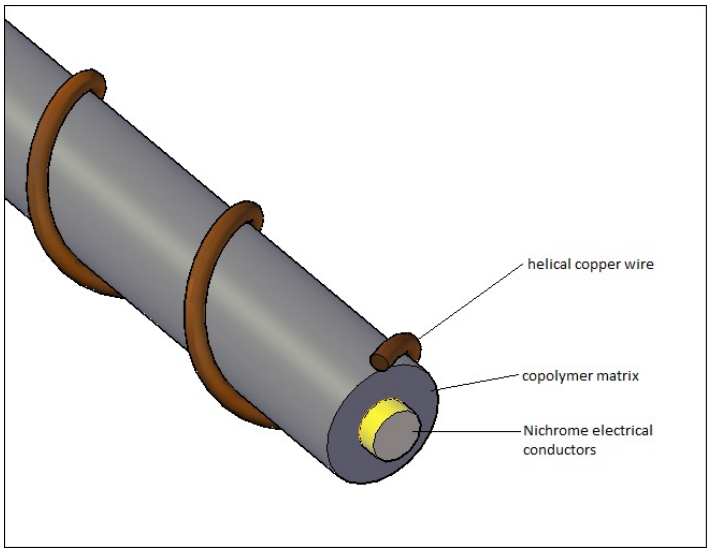

Figure 2. Distributed sensor featured.
Source: Author's own elaboration.

In order to detect and locate a milk leak, we designed our distributed sensor with two electrical conductors isolated by a copolymer (which will be introduced in the next section). One conductor runs along the central axis of the cylindrical-shaped sensor, inside a copolymer matrix, while the other is arranged in a helical manner, in tight contact with the external surface of the copolymer layer. The helical formation gives sustenance to the sensor, covering all the possible angles of liquid escape.

Materials and methods

Materials

The monomers used: poly(acrylic acid) (> 98%) and poly(n-butyl acrylate) (> 98%), provided by Aldrich were purified by 80 °C temperature vacuum distillation. The initiators potassium persulfate (> 99%) and ammonium persulfate (> 99%), supplied by Sigma Aldrich, were purified by recrystallization and vacuum dried at 35 °C. Polyvinyl alcohol (>80% hydrolysis, JT Baker), sodium lauryl sulfate (>99%, JT Baker), and Triton X-305 (70% w/w solution, JT-Baker) were used as surfactants. For detecting and locating leaks, pasteurized whole milk was used with an average fat content of 3.28 g per 100 ml provided by Grupo Lala S.A. de C.V. The acquisition data hardware was formed with an array of resistances of 22 ohms +/- 1% accuracy, connected in series at the beginning and end of the laying of the sensor. To monitor brownouts, an acquisition card USB-6009 analog input data with 14-bit resolution and a sampling rate of 48 kS/s provided by National Instruments™ was used.

Copolymer synthesis

The copolymer described was synthesized by radical emulsion polymerization with 30% w/w of hydrophilic component (acrylic acid) and 70% w/w of hydrophobic component (butyl acrylate). The copolymer resulting from the synthesis had a conversion degree of 99.11% (Mendoza-Payán, 2011).

The equipment used to perform the synthesis consisted of three neck flasks with mechanical stirring generated with a Teflon impeller attached to a stainless-steel drive shaft, a cooling tube, and a burette for adding ingredients. The system was tightly sealed to prevent the entry of oxygen and the escape of monomers; an isothermal water bath was used to increase the temperature.

The reaction system was initially stabilized at 70 °C and 350 RPM stirring rate, besides adding nitrogen by bubbling in order to purge the remaining oxygen within the system, containing initially only butyl acrylate dispersed in surfactants (oil). Subsequently, half of the dissolved initiator was added in 20 ml of water for a period of 5 min; after this period, an aqueous solution of acrylic acid with other initiators was added over a period of 90 min, the polymerization time was set at 4 h. The reaction that occurs during the polymerization process is as follows (Figure 3).

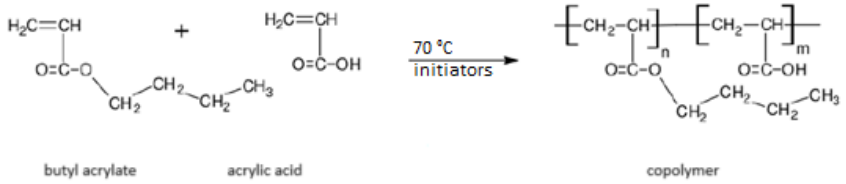

Figure 3. The general reaction of the polymerization process.
Source: Author's own elaboration.

A latex with a diffusion coefficient of 5.89E-05 cm^2/s (Mendoza-Payán, 2011) was obtained from the polymerization process, which was dried in a convection oven at 90 °C for 24 h.

The product obtained was cut into small pieces to homogenize it with polyethylene glycol of molecular weight equal to 2 000 gr, which was used as an internal lubricant (plasticizer) to improve integration of the material during the extrusion process. The mixing (performed in a Brabender© plasticorder using Banbury blades) was conducted for a period of 10 min, using a weight ratio of plasticizer of 2% relative to the total mass of the copolymer.

The coextrusion process of the sensor cable prototype was performed in a laboratory extruder Brabender using a die coater cable and, as core employment, nichrome wire (an alloy of nickel and chromium) unprotected outer resin AWG 28. The line production was mounted on a pulling system and in extrusion conditions, shown in Table 1. Subsequently, the prototype wire obtained was wrapped spirally (windings) by a copper wire 28 gauge (AWG 28) using a continuous automatic winding, achieving a distribution of copper wire along the sensor cable with a distribution of 1.2 cm/cycle.

Table 1. Extrusion equipment parameters.

Parameter	Value	Unit
Feed temperature	65	°C
Plasticizing temperature	70	°C
Compression temperature	75	°C
Dosage temperature	80	°C
spindle speed	3	RPM
spindle L/D	20	Dimensionless
spindle torch	7.2	N.m

Source: Author's own elaboration.

Experimental methodology

Considering that this is a resistive sensor, a voltage feed is necessary to measure resistance changes with these variations and to determine if a leak is detected in addition to the calculations relevant for leak location through equation 1. Figure 4 shows the system composed of a closed sensor circuit where 24 VRMS (root mean square voltage) is applied to the helical copper (wire 1). The Nichrome core (wire 2) is conditioned for current return, the current measurement system is positioned at the ends of the sensor. The values are acquired at a sampling rate of 24 data per minute. The system contains an A/D data acquisition card (Katalin, 2006), capturing the voltage drop across a pair of 22 ohm precision resistors, one for each channel, and calculating from this the current flowing through that section of the circuit. In the first 5 minutes, the system does not come into contact with any liquid, this stage is to obtain basal values of the system without the presence of liquids. At the end of this step, a section of the sensor circuit (about 10 cm) is flooded in whole milk, simulating a leak; this stage is extended for a period of 50 minutes. After this period of time, the liquid is removed, allowing the desorption stage to take place, which takes 60 min, with a total of 2 h of testing. The test is performed at 25 °C and 50% relative humidity.

$$L_C = (I_2 * L_T)/(I_1 + I_2) \quad (1)$$

where L_C sensor is the length in which the leak is presented, L_T is total length of the sensor, I_1 is channel 1 current value, and I_2 is channel 2 current value.

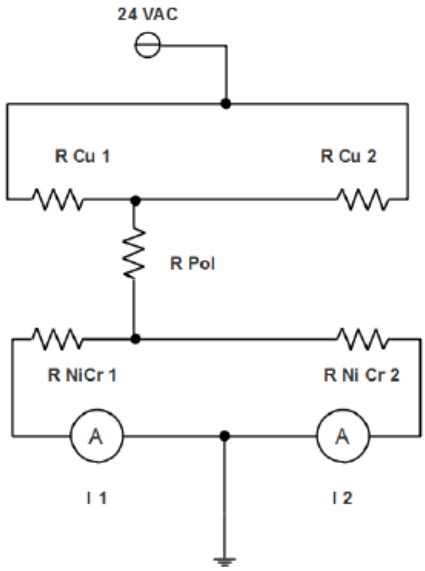

Figure 4. Sensing system equivalent circuit.
Source: Author's own elaboration.

Results

Figure 5 shows the behavior of the intensity of the sensor that was exposed to milk sample simulating a leak. In the first 900 seconds the intensity is close to 0 amps. When milk makes a bridge, the value immediately increases by about 0.01 amperes in both intensity meters; subsequently, the value slowly increases to 0.024 A. After 50 minutes, the sensor is retired from the milk container; then, the intensity goes down with a characteristical desorption behavior. Fahim *et al.* (2017) presented a sensor system that needs several seconds to read the leak of methanol using the simple threshold technique.

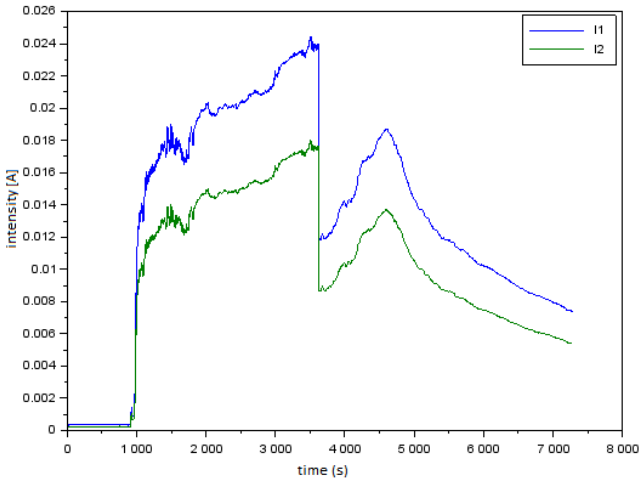

Figure 5. Sensor typical intensity response in a milk leak situation.
Source: Author's own elaboration.

Figure 6 shows the resistance response of the sensor to the presence of a leak milk sample; the three sections can be clearly seen. The first 10-minute section corresponds to the baseline, the second is a reduction of resistance in response to the presence of liquid during a 50-minutes lapse, and the third is the desorption phase.

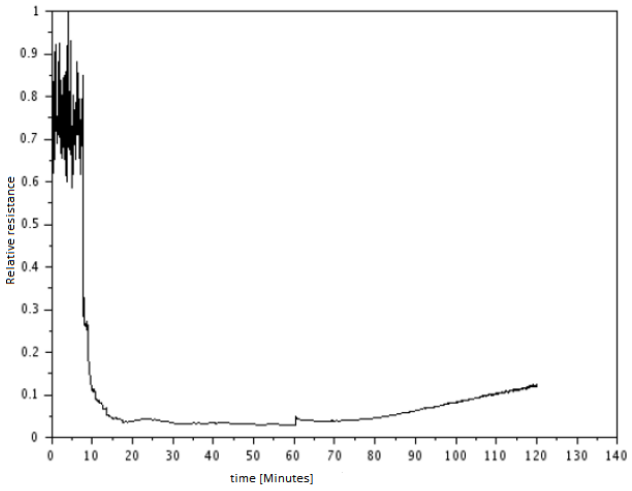

Figure 6. Sensor typical resistance response in a milk leak situation.
Source: Author's own elaboration.

The proportion of monomers significantly influences the properties of copolymers. The high butyl acrylate concentration (70%) in polymer synthesis provides flexibility and improves the adsorption-desorption characteristics of the polymer (Zhou *et al.*, 2014), whereas poly(acrylic acid) (30%) gives polymer cohesion features (Colombani *et al.*, 2007).

This response is repeated for both sections because, as shown in the equivalent circuit leakage, it generates an electrical current partition to both ends of the sensor and with these two values under equation 1 the position of the leak can be calculated. The leak location is very precise because the nichrome used has a resistance of 13.69 ohms per meter. The noise present in the signal is overcome by the current intensity, which is due to the increased conductivity of the electrolyte. The precise location of the leak can be seen in Figure 7 (2.07 m on the y-axis). Cardell-Oliver (2015) found the location of the leakage by triangulation using the different time-of-arrival methods of different sensors in the pipe network, but the researcher did not find the precise location of the leak.

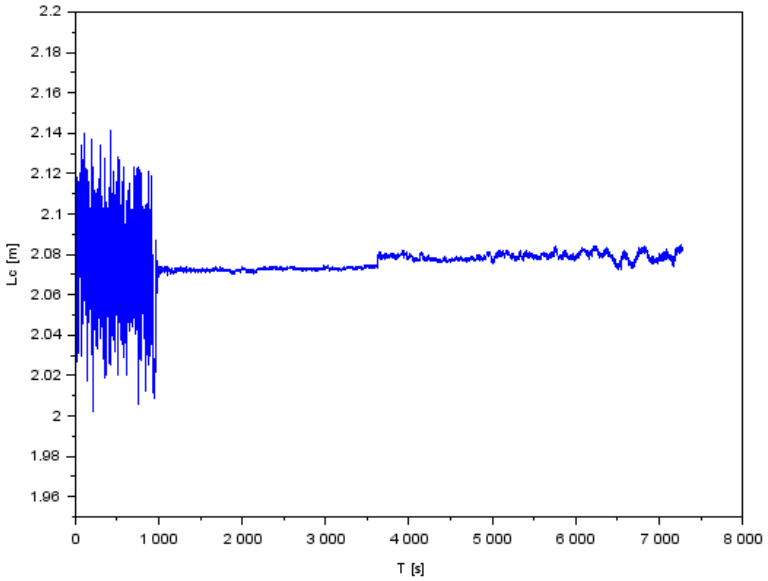

Figure 7. System development for leak localization.
Source: Author's own elaboration.

Figure 7 shows the electrical behavior of the sensor on its internal circuit C2. While the sensor does not enter in contact with milk (or another aqueous suspension), the resistivity of the copolymer layer is very high. Then, the current passing from wire 1 to wire 2 is very low. Indeed, during the first five minutes of the test, when the sensor is dry, there is almost no current in this circuit. However, when milk is added to the test container, the current intensity measured in the ammeter number 1 starts to increase in less than three minutes. As expected, this increase rises very fast in the first 10 minutes, and then the current grows more slowly and in a bluff fashion. This last trait is due to the irregular diffusion of the milk inside the sensor (spatially of its water fraction). At the end of the test, at 60 minutes, the sensor is removed from the test container. A drastic drop in the current intensity is noted, from 5.2 mA to 2.7 mA, that is, 48%. Unexpectedly, there is a recovery in the following minutes, to 3.9 mA. This can be due to the diffusion inertia of the milk inside the sensor as well as to a realignment of the structure of the sensor. After that, the electric current continues to fall until the initial value. In Figure 8, the data is shown until 120 minutes because a total recovery takes almost 24 h.

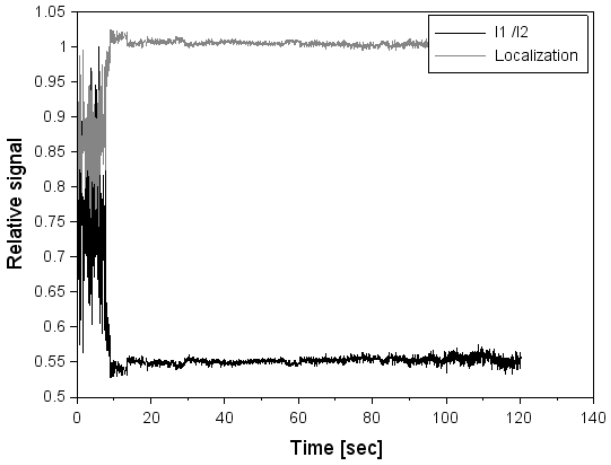

Figure 8. The behavior of the sensor in a milk leak (Intensity units).
Source: Author's own elaboration.

The sensor works thanks to the short circuit produced between both conductors, due to the diffusion of milk water fraction inside it. Therefore, it is important to evaluate with the most difficult product to diffuse, such as whole milk. Hence, if the sensor works with it, then it will work with any other milk. Figure 9 illustrates the result of a thermo-gravimetric analysis performed to whole milk at 100 °C (Thermogram from a TGA equipment, Model 2950, TA Instruments). In the first 20 minutes, water fraction evaporates, then fat degradation starts. The fat percentage is about 6%. Therefore, the water fraction of this milk is around 94%, which is high enough to ensure good sensor function.

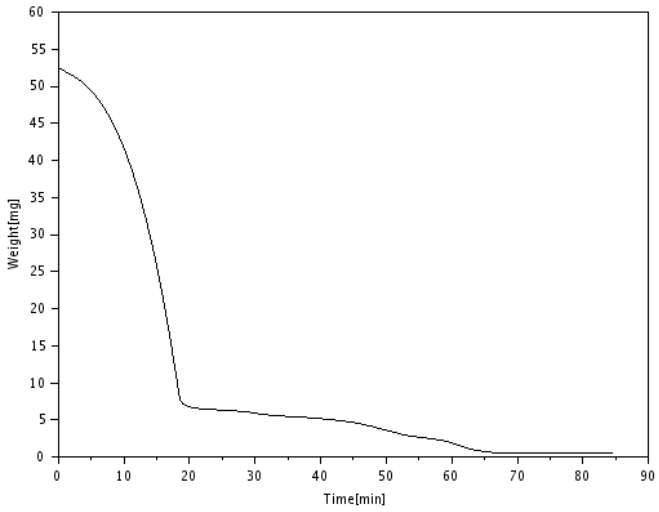

Figure 9. Thermal gravimetric analysis of raw milk.
Source: Author's own elaboration

Discussion

From Figure 5, 6, and 7 we can deduce that in the first initial five minutes, the operation of the sensor without the existence of leakage, currents are low due to the high resistance of the polymer, adding two features to the system. The first feature is a detection threshold, where a minimum current is correlated to determine the existence of a leak; when the current surpasses this threshold, the system begins with the calculations of location. Wu *et al.* (2017) designed a leak sensor that moves inside the pipe to travel to the leak point and needs constant fluid velocity. Our design does not need to travel. The second feature is based on energy savings, since the system consumes little power in the dry state (0.4 mA). The response phase after 5 min of contact of the sensor with the milk generates a sufficiently high and stable current in amperes to determine the location of the leak with good accuracy. In the last stage the slope of desorption is low, requiring several hours for the sensor to recover the dry state. This phase belongs to the liquid desorption, which shows slow recovery behavior, partly because it requires liquid evaporation; and since the polymer sensor serves as a means of water removal with respect to milk fat, then a concentrated layer of milk fat is formed, slowing evaporation. Regarding diffusion, a coefficient must have a value between 10 m^2/s – 12 m^2/s, which depends largely on temperature (Salas *et al.*, 2018). The sensors produced by Wu *et al*. (2017) need to travel 0.4 m/s to find the leak; if the flow rate varies, then the location calculation will be wrong. The channels' current levels must overcome the time lapse detection of 5 min of contact with the liquid, after this time, the leak location accuracy has error levels of +/- 1%. Jia *et al.* (2014) used a hoop strain system to localize the distances of the leakage positions with an average error of 7.33%.

Conclusions

This system has the ability to detect and locate leaks or micro leaks in pipeline systems of a pasteurization and bottled milk plant with good accuracy (+/- 1%). Another characteristic of the proposed polymer is that it is reusable; once the recovery time (desorption phase) has passed, it continues to function. The system's safety is quite high since the voltage and current levels with which the system works are low, so there is no risk of electrocution. Regarding chemical composition, the system components are of low or zero toxicity.

Declaration of interests

The authors declare that they have no conflict of interest.

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